Chapter 62 - **GALLERY OF SANS DATA IMAGES**

This author has collected, over the years, a gallery of SANS data images from oriented samples. Some of these images are included here purely for their esthetic (artistic) value. A reference has been included in each case. This reference does not necessarily include the same SANS images, but is representative.

1. SHEARED MULTI-LAYER VESICLES

Multilayer vesicles (MLV) have an onion-skin type of structure. AOT surfactant in brine (i.e., salty) water solution forms MLVs. When sheared, MLVs yield characteristic SANS images dominated by orientation of the lamellae (Bergenholtz-Wagner, 1996). Couette shear is effective at orienting the lamellar structures. Oscillatory shear produces more orientation than simple shear. The tangential view (whereby the neutron beam is parallel to the shear direction) measures the shear gradient and neutral directions.

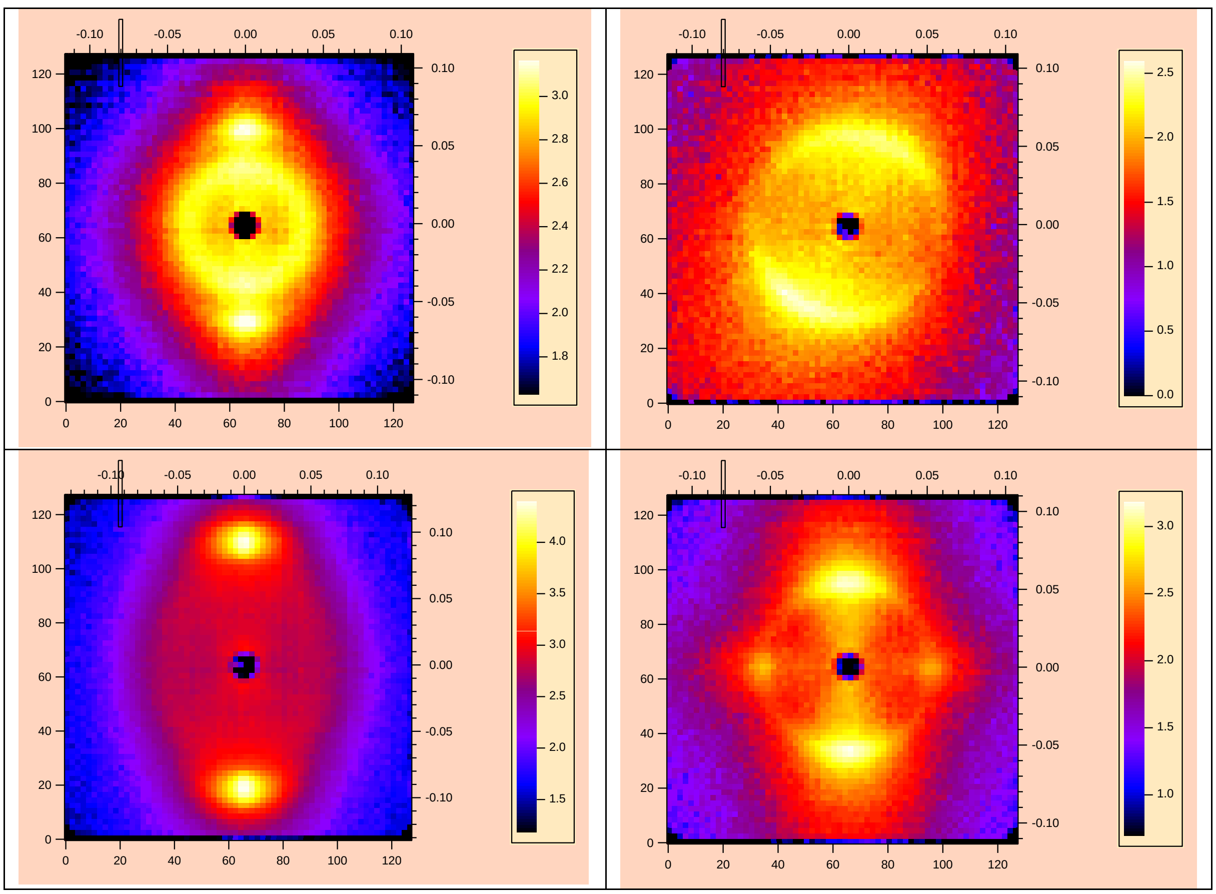

Figure 1: AOT in brine/D_2O multilayer vesicles sheared in a Couette shear cell. Top left: radial view under simple shear and low shear rate (0.025 rps), Top right: tangential view under simple shear and high shear rate (7.0 rps), Bottom left: radial view under oscillatory shear and high shear rate (15 rps), Bottom right: tangential view under

oscillatory shear and high shear rate (15 rps). The two SANS data sets shown in the bottom were taken at high temperature (50 ^oC).

2. THE BUTTERFLY PATTERN

SANS measurements are made in reciprocal space. When samples are oriented along the horizontal direction, they yield SANS patterns oriented along the vertical direction. This is due to the fact that reciprocal Q space and direct space form a conjugate pair. The exception to this is the case of the so-called "butterfly" pattern whereby orientation in direct space and in reciprocal space are along the same (here horizontal) direction. Cross linked polymer networks are characterized by a butterfly scattering pattern. Here, a SANS image from a crosslinked and stretched poly(dimethyl siloxane) gel is included (Mendes et al, 1996).

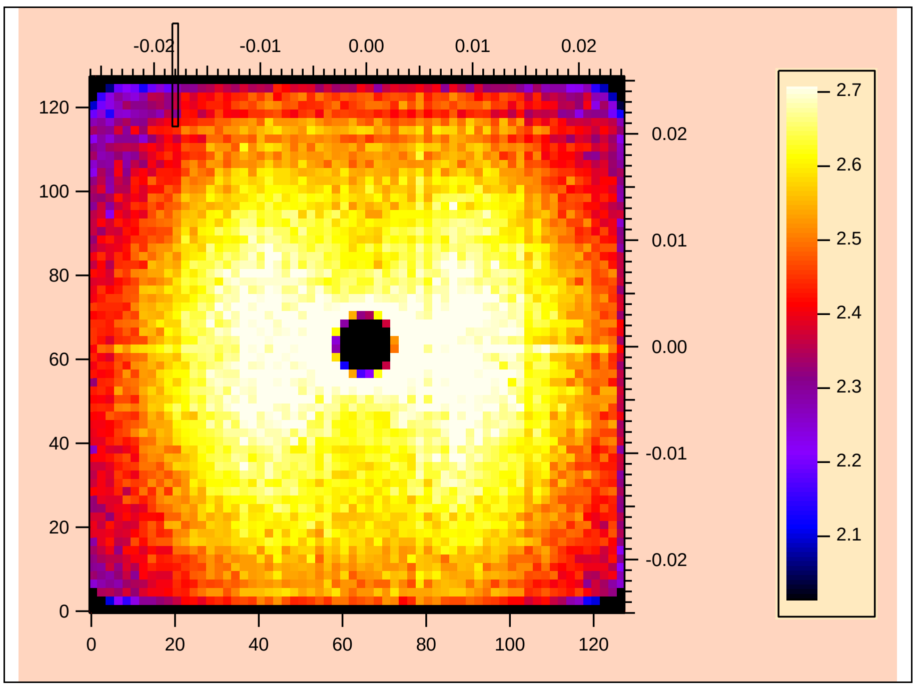

Figure 2: Butterfly SANS pattern from stretched poly(dimethyl siloxane) gel consisting of a mixture of crosslinked and (deuterated) linear polymer chains.

3. PACKED SPHERES

Highly packed silica particles in D_2O solution can form a "single crystal" texture characterized by bright diffraction spots under gentle shear (Butera et al, 1996). The SANS image shows 6-fold symmetry pointing to a cubic structure (body centered cubic). Four orders of diffraction spots are visible before the instrumental smearing becomes overwhelming (at high-Q).

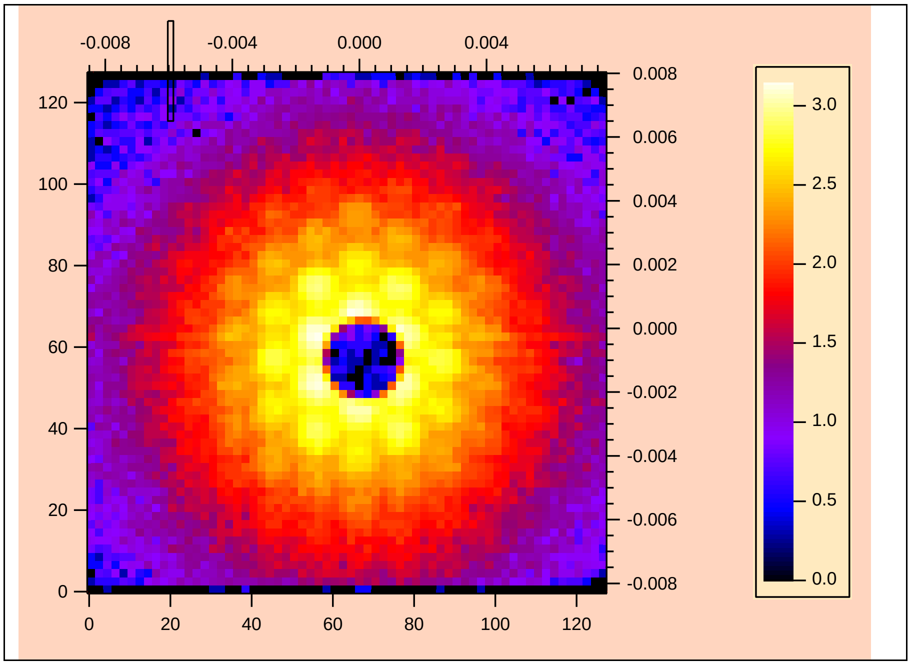

Figure 3: Single crystal diffraction pattern obtained from highly packed silica particles under gentle shear and in D_2O.

4. MULTI-PHASE ALUMINUM TEXTURE

SANS diffraction pattern obtained from a multi-grain aluminum sample is shown. Three major grains can be resolved. Note the dark blue spot on the middle-left part of the image. This is a damaged spot on the neutron detector produced by neutron over-exposure.

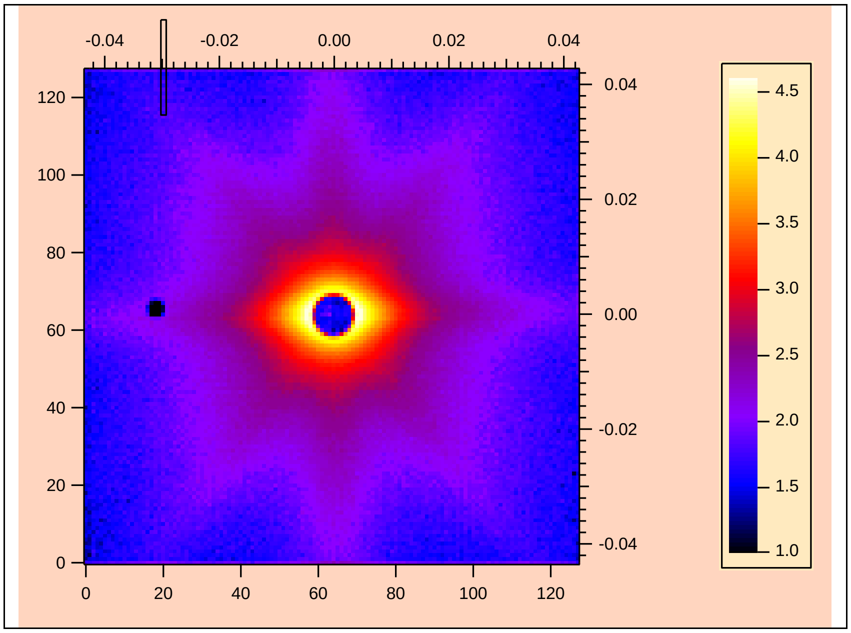

Figure 4: SANS diffraction pattern from multi-grain aluminum.

5. KANGAROO TAIL TENDON

Collagen from kangaroo tail tendon is a highly oriented fiber with crystalline structure along the fiber. Five order reflections can be resolved. Note that the second order reflection is extinct.

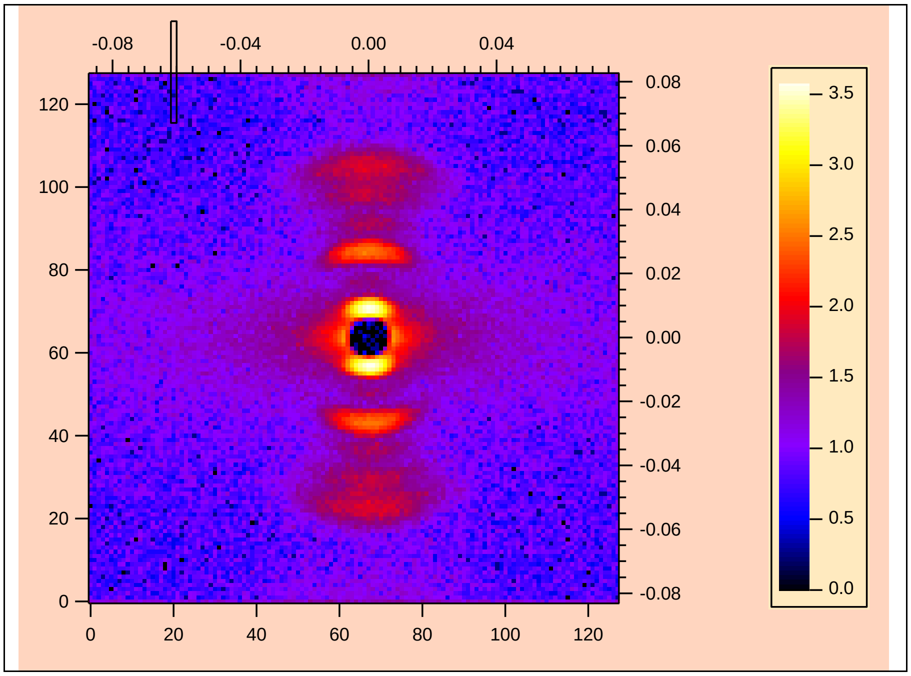

Figure 5: Scattering pattern from collagen from a kangaroo tail tendon showing the strong first and third reflection peaks as well as weak higher order reflection peaks; the second reflection peak is not allowed. The ordered structure is along the fibers and has a d-spacing of 667 Å.

6. TWINNED CRYSTAL

SANS from a twinned single-crystal of $NdBa_2Cu_3O_7$ (high T_c superconductor) is shown at 100 K. The oxygen content can be changed from O_7 to O_6. The O_6 system is antiferromagnetically ordered, tetragonal, and insulating, while the O_7 system is orthorhombic, and is superconducting (Tc around 90 K). The twinned crystal grew along two orthogonal directions. Crystal boundaries occur when two crystals inter-grow with a highly symmetrical interface, often with one crystal being the mirror image of the other; atoms are shared by the two crystals at regular intervals. The twinning was produced by the tetragonal to orthorhombic distortion. Scattering is mostly from nuclear (not magnetic) scattering. SANS data for a related system $YBa_2Cu_3O_7$ have been published (Keimer et al, 1993).

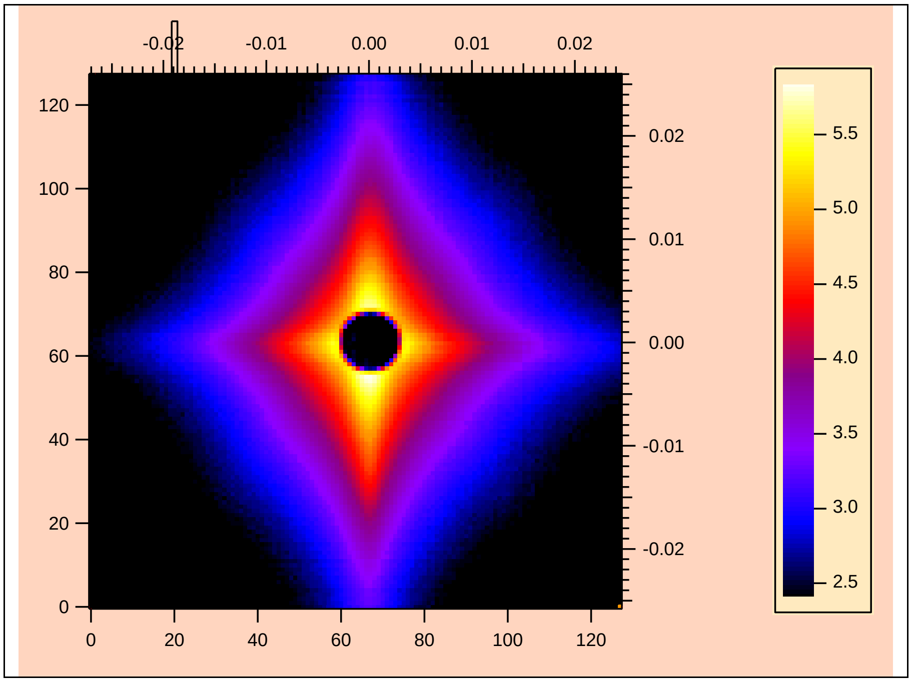

Figure 6: SANS data from the high T_c superconducting $NdBa_2Cu_3O_7$ cuprate at 100 K. The oriented structures characterizing the two crystals forming the twinned crystal are orthogonal (i.e, have orthorhombic symmetry) yielding the cross-like SANS patterns.

7. CORRELATIONS IN A MULTIPHASE COMPOSITE

SANS data image taken from a multiphase aluminum oxide composite (Adolphs et al, 2002) is included. Distortion of the structure is observed. Further details are not available.

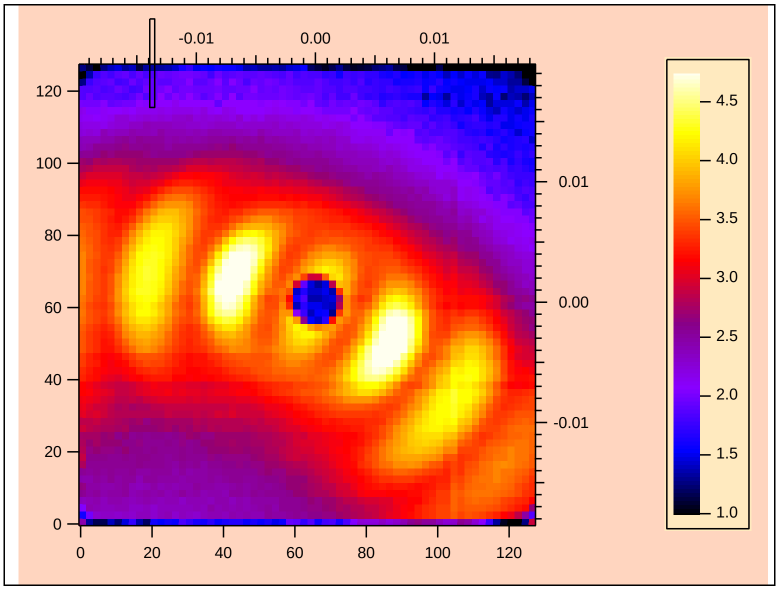

Figure 7: SANS from a multiphase aluminum oxide composite.

8. SHEARED SPHERICAL MICELLES

Anisotropic SANS data from P85 Pluronics micelles sheared in a Couette shear cell are shown. The characteristic hexagonal peak pattern (six fold symmetry) points to a cubic structure formed by the spherical micelles for 25 % mass fraction P85 in D_2O solutions. P85 is a triblock copolymer of poly(propylene) which is hydrophobic as the middle block and poly(ethylene oxide) which is hydrophilic as the outside blocks (PEO-PPO-PEO). P85 micelles are well formed at ambient temperature. Shearing helps the packing of the spherical micelles into a face centered cubic structure (Slawecki et al, 1998).

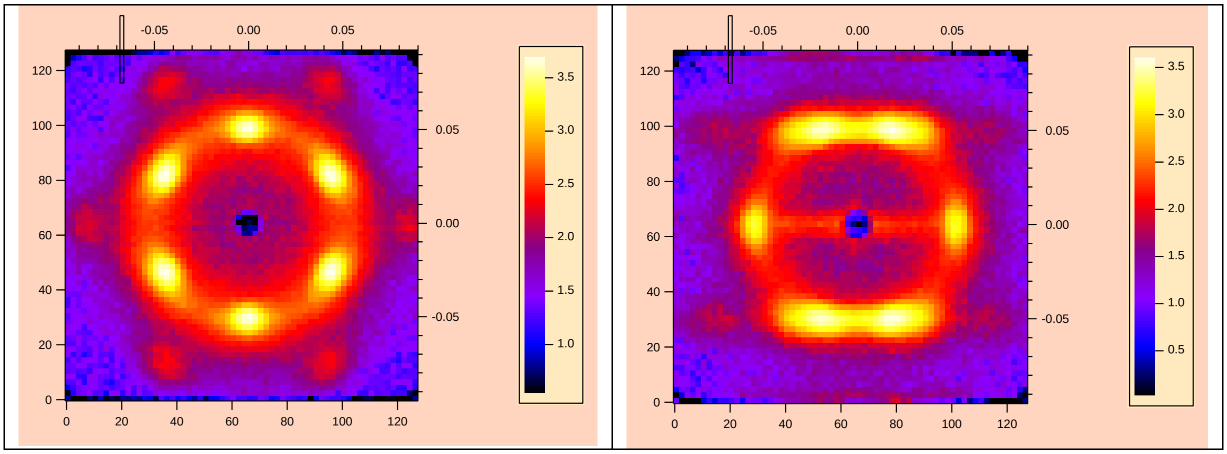

Figure 8: SANS data from 25 % P85 Pluronic (PEO-PPO-PEO triblock copolymer) in D_2O under Couette shear (5 Hz frequency) at 40 ^oC. The micelles form a cubic "single crystal" structure. Left: radial view. Right: Tangential view.

9. PEPTIDE ORIENTATION IN MEMBRANES

Peptides that are embedded in membranes produce highly oriented structures and yield a good harvest of interesting SANS images. Two antibiotic peptides (alamethicin or magainin) were investigated extensively. These were oriented between quartz plates and embedded into phospholipid bilayers forming the membrane. Deuterated water fills the inter-layer space for enhanced neutron contrast. Peptides form inter-layer “pores” that can be clearly observed. Temperature and relative humidity were controlled in order to monitor hydration effects on the structures. Fully hydrated samples show no inter-layer correlation. Dehydrated samples show strong such correlation that shows up as rich anisotropy in the SANS pattern characteristic of “single crystal” structure. In order to sample both the in-plane and the out-of-plane structure, the oriented membranes were tilted with respect to the neutron beam (Yang et al, 1998; Yang et al, 1999).

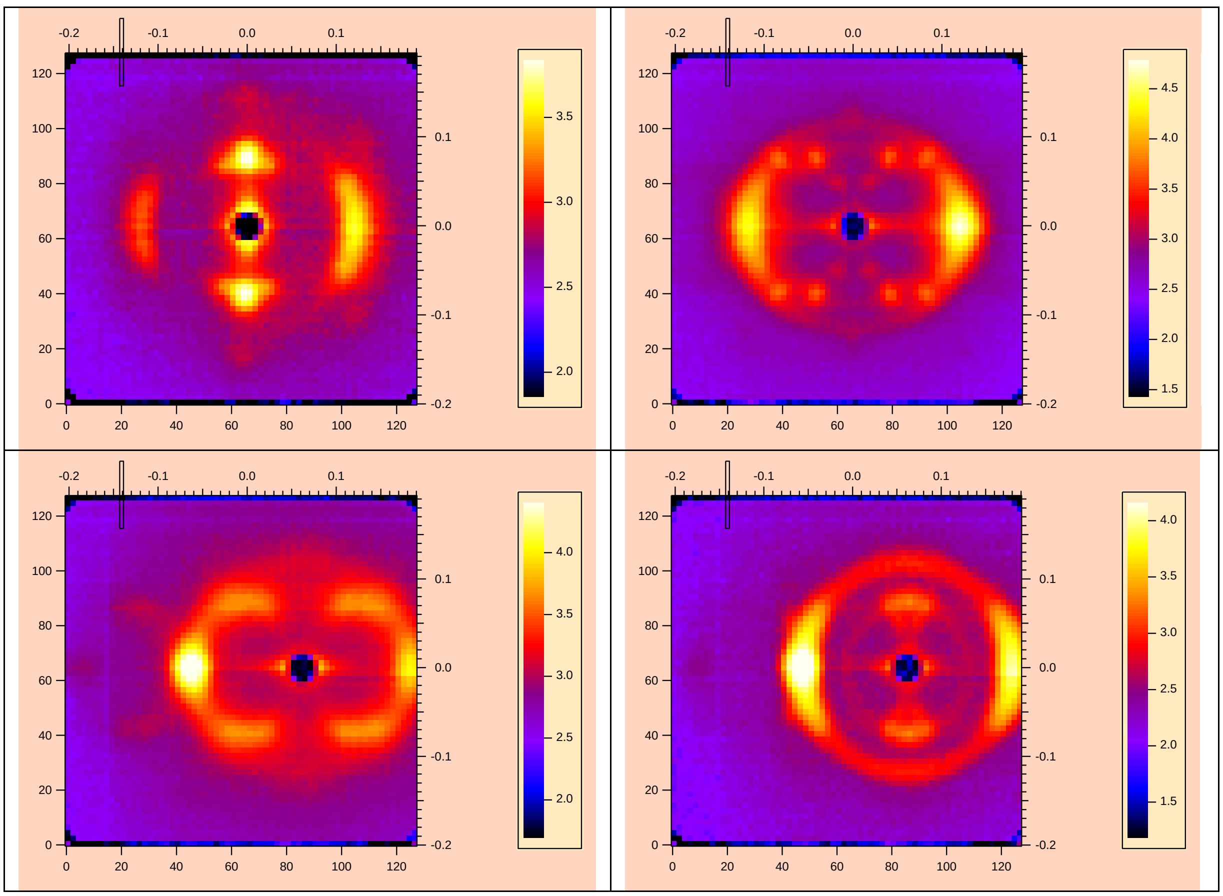

Figure 9: SANS data from peptides embedded into membranes and oriented between quartz plates. The sample was oriented at 60 ^{o} to the neutron beam in order to observe structures both parallel and perpendicular to the membrane surface. Top left: 60 ^{o} alignment angle. Top right: 80 ^{o} alignment angle. Bottom left: 80 ^{o} alignment angle and different hydration level. Bottom right: -80 ^{o} alignment angle. All patterns were obtained at 28 ^{o}C sample temperature but with different hydration levels.

These images have a number of bright spots and more smeared diffuse features. The interlayer spots can be easily distinguished (specular scattering) since these are the brightest. The other spots and diffraction features are from the peptide structure. The major elements of that structure can be resolved based on the various clues available. It looks like there are two characteristic d-spacings throughout; one from the inter-layer spacing and one from the inter-peptide nearest neighbor (first coordination shell) d-spacing. This field of research has barely started (Yang et al, 1998; Yang et al, 1999).

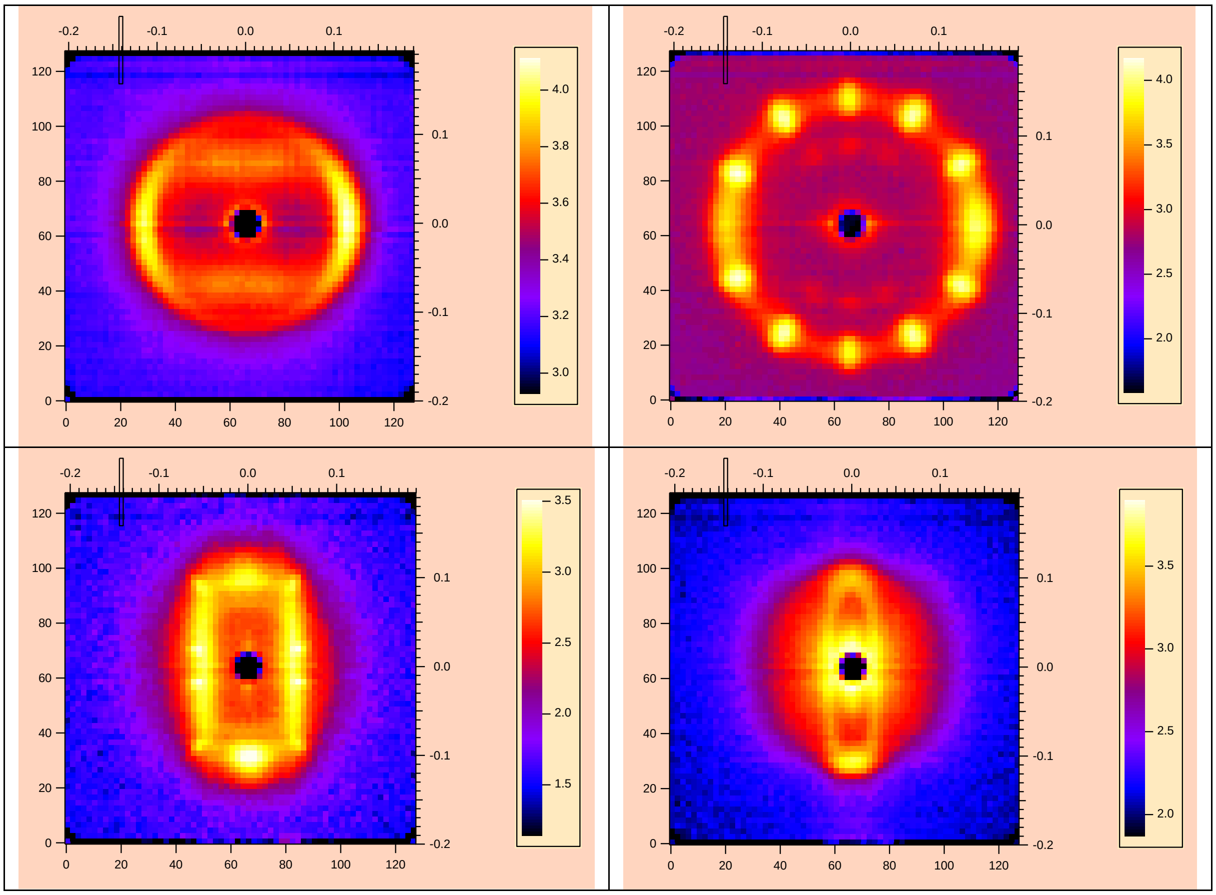

Figure 10: More patterns obtained from peptides embedded into membranes and oriented between quartz slides. The main features are understood. These structures, however, have not all been resolved yet.

Once the observed structures have been resolved in detail, one could think of using partially deuterated blocks within the peptides to nail down these structures in more detail. Nowadays, using deuterated amino acid sequences to synthesize specific peptides is possible.

REFERENCES

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QUESTIONS

1. When is the "butterfly" pattern obtained?
2. A diffraction pattern containing six-fold symmetry points to what possible structure?
3. What is the difference between "single crystal" diffraction and "powder" diffraction?
4. Fiber diffraction is characterized by what type of pattern?

ANSWERS

1. The butterfly pattern is obtained when the direction of orientation of the iso-intensity contour plots is the same as the orientation of the anisotropy in the sample.
2. The cubic and the hexagonal structures are characterized by six-fold diffraction patterns.
3. Single crystal diffraction is characterized by spots while powder diffraction is characterized by diffraction rings.
4. Fiber diffraction is characterized by spots aligned along one direction.